Effects of interfacial modification on diamond film adhesion

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ABSTRACT

Microlithographic surface patterning has been investigated as a means of improving diamond film adhesion on noncompatible substrates. This technique produces significant improvements in film adhesion beyond identical unpatterned substrates, although sufficient film stresses can develop to induce subsurface substrate fracture. The substrate etching geometry can be chosen to simultaneously produce an antireflective surface relief.

1. INTRODUCTION

Polycrystalline diamond thin films offer a unique combination of physical properties highly desirable for optical device applications: high hardness, wideband transparency, chemical inertness, and high laser damage thresholds. Application of these coatings to optical device structures, however, is often impeded by poor film adherence and surface roughness. While films of substantial thickness can be produced routinely on Si, films grown on numerous other optical materials under equivalent conditions often delaminate at thicknesses of only a few microns due to lack of interfacial chemical bonding, intrinsic growth stresses and thermal expansion mismatch. To fully exploit the potential of the diamond films in optical applications, fabrication on less-compatible substrates must be addressed. Additionally, the highly faceted polycrystalline diamond films cause high levels of scattering and normally must be polished to achieve good optical transmission.

One approach to improving diamond film adhesion is the modification of the original film-substrate interface. In this work, the use of microlithographic patterning of the substrate surface has been investigated as a means of improving diamond film adhesion. Surface patterning has been shown to allow control of film microstructure,[1] and the current work demonstrates that it may also provide significant improvements in film adhesion and infrared transmission.

2. EXPERIMENTAL

For these experiments, fused silica and Ge substrates were used. The substrates were patterned by standard photolithography and dry etching techniques, producing a grid pattern of etched lines approximately 1 μm deep. The line widths were 1-3 μm, with center-to-center separations of 10-15 μm. Diamond films were grown using microwave plasma CVD with a source gas composition of 0.5% CH_4, 0.2% O_2, and the balance H_2. Substrate growth temperatures of 750°C were used for the SiO_2 and 550°C for the Ge as determined by optical pyrometry. Unpatterned substrates were included in the depositions for experimental comparison, with all samples given identical diamond polish pretreatments to enhance film nucleation.

3. RESULTS AND DISCUSSION

Growth of the diamond film on the patterned SiO_2 was continued up to a thickness of approximately 24 μm. By contrast, the film on the bare substrate grown simultaneously began to delaminate after thicknesses of approximately 1-2 μm, demonstrating the adhesion improvement associated with the microlithographic patterning. The patterned film surface continued to

exhibit the original substrate topology even after these thicknesses (Fig. 1). Also evident in Fig. 1 is the appearance of surface cracking. Cross sectional examination in the scanning electron microscope (SEM) revealed that these were not due to film delamination, but rather were associated with subsurface substrate fracture (Fig. 2). The interfacial integrity of the film was maintained even at stresses sufficient to induce substrate fracture.

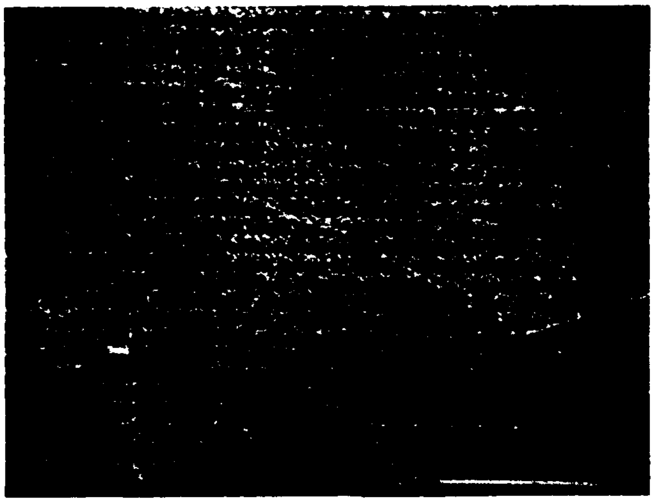

Fig. 1. Surface morphology of diamond film grown on etched SiO_2.

Fig. 2. Cross sectional SEM micrography illustrating subsurface fracture in SiO_2 substrate.

Similar results were obtained for growth on the patterned Ge substrates. Film adherence on the patterned substrate was maintained to thicknesses beyond 30 μm, while on the unpatterned substrate delamination occurred at approximately 1 μm. As observed with the SiO_2, the original surface pattern was maintained in the film even at substantial thicknesses (Fig 3). Ultimately, however, sufficient stresses were generated by the growth process to induce substrate fracture, with the original film-substrate interface still intact after fracture.

Fig. 3. Surface morphology of diamond film grown on etched Ge.

It is well established that the diamond films, with their high modulus and low thermal expansion coefficients, can generate significant stresses.[2] This can induce substantial curvature to bulk substrates, or even lead to substrate fracture as seen here. This type of subsurface fracture by a thin film has been modeled by Drory et al.[3] In their analysis, both shallow and deep cracks will be generated in the substrate parallel to the film interface, with the crack depths related to the film and substrate thicknesses and elastic properties. Using the appropriate values for the diamond film on SiO_2, one arrives at theoretical crack depths consistent with those experimentally observed.

The use of substrate surface patterning, while improving diamond film adhesion, will impact the optical performance of the device by the introduction of interfacial scatter. Modification of the pattern geometry can be made, however, to minimize this effect. Comparison has been made between the original grid pattern and a square array of etched holes of similar spacing. The array of holes reduces the area fraction being etched, significantly reducing the scatter losses in the short wavelength infrared without sacrificing the adhesion enhancement of the grid pattern. An example of the diamond film morphology on Ge produced in this manner is shown in Fig. 4.

An alternate approach may be taken to mitigate the optical scatter losses of the surface etching. Here, the etched geometry can be chosen small enough relative to the design wavelength to prevent diffractive losses. In this manner, a fine-scale etched surface relief which progressively grades the effective index of refraction between that of air and the Ge substrate can be generated. This type of structure, commonly referred to as a "moth eye" surface, can reduce Fresnel reflection losses to provide antireflective properties.[4] Such an antireflective surface relief structure has been fabricated using a polycrystalline diamond film grown on an etched Ge substrate (Fig. 5).[5] By the appropriate choice of the etching geometry and diamond film thickness, transmission levels in excess of 95% can be obtained (Fig. 6).

4. CONCLUSIONS

Microlithographic patterning of substrate surfaces prior to diamond growth provides a means of improving film adhesion beyond that of nonpatterned substrates. This offers a potential method of diamond film growth on incompatible or poorly compatible substrates.

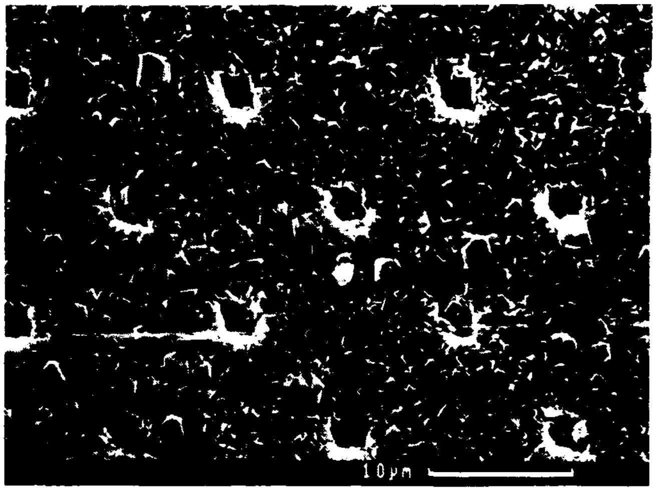

Fig. 4. Diamond film grown on Ge substrate using etched hole array for adhesion enhancement.

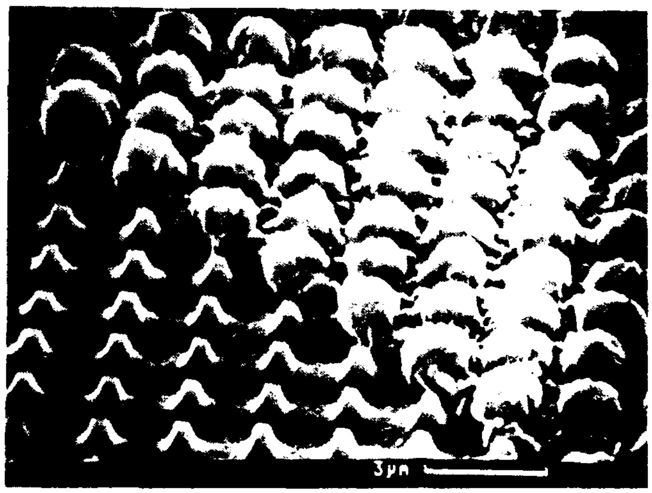

Fig. 5. SEM micrograph of diamond film antireflective surface on Ge.

Implementation of this technique for thick diamond films will require considerations of the large stresses that may develop during film growth. For these cases the film stresses may exceed the substrate fracture stress, causing subsurface failure. Certain regimes of temperature and hydrocarbon content which produce reduced stresses in the diamond films have been identified,[2] and growth under these conditions may permit thicker film development on the patterned substrates. By appropriate choice of the etching geometry, this technique can be further extended to the fabrication of diamond antireflective surfaces.

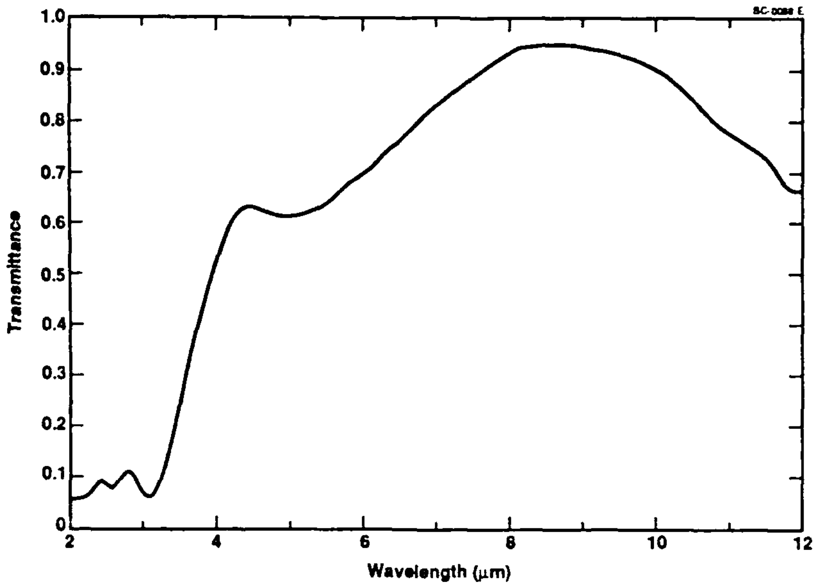

Fig. 6. IR transmission spectra of Ge substrate with diamond antireflective surface. Rear surface coated with dielectric multilayer.

5. ACKNOWLEDGMENT

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6. REFERENCES

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